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] ; T	TRANSMITTAL LETTER TO THE UNITED STATES		Mo6994/LeA 33,570	
	DESIGNATED/ELECTED OFFICE (DO/EO/US)		U.S APPLICATION NO (If known, see 37 CFR 1.5	
	CONCERNING A FILIN	NG UNDER 35 U.S.C. 371	To be Assigned 882 1 6	
INTERN	NATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED	
	P00/08605	04 September 2000 (4.09.00)	13 September 1999 (13.09.99)	
	OF INVENTION bonate Slabs			
		Y, Ruediger; ANDERS, Siegfried; RC	THMER Juergen: KUEHLING	
Steffen;	; LANZE, Rolf; PREIN, Micha	ael; NEUMANN, Rainer and KAUTH	H, Hermann	
Applican	nt herewith submits to the United Sta	ates Designated/Elected Office (DO/EO/US)	the following items and other information:	
1. X 7	This is a FIRST submission of items	s concerning a filing under 35 U.S.C. 371.		
2. 🔲 Л	This is a SECOND or SUBSEQUE !	NT submission of items concerning a filing u	inder 35 U.S.C. 371.	
		ational examination procedures (35 U.S.C. 37		
i	items (5), (6), (9) and (21) indicated	below.	• • • • • • • • • • • • • • • • • • • •	
		iration of 19 months from the priority date (A	rticle 31).	
	A copy of the International Application			
_	 a. [X] is attached hereto (required) b. has been communicated by 	d only if not communicated by the Internation	nal Bureau).	
			ena Offica (DO/LIS)	
		ication was filed in the United States Receiving	,	
_	a. x is attached hereto.	the International Application as filed (35 U.S.	C. 3/1(c)(2)).	
	. =	itted under 35 U.S.C. 154(d)(4).		
		ernational Aplication under PCT Article 19 (3	35 U.S.C. 371(c)(3))	
	`	ed only if not communicated by the Internation		
_		by the International Bureau.	Mui Bureau,	
	_	ever, the time limit for making such amendme	anto has NOT avnired	
	d. have not been made, nowe		nts has 1401 expired.	
			: 12/25112 2 251/ \/2\)	
		he amendments to the claims under PCT Artic	cle 19 (35 U.S.C. 3/1 (c)(3)).	
	An oath or declaration of the invento			
	An English lanugage translation of the Article 36 (35 U.S.C. 371(c)(5)).	he annexes of the International Preliminary Ex	xamination Report under PCT	
Items	s 11 to 20 below concern document	t(s) or information included:		
11.	An Information Disclosure Stateme	ent under 37 CFR 1.97 and 1.98.		
12.	An assignment document for recor	rding. A separate cover sheet in compliance v	with 37 CFR 3.28 and 3.31 is included.	
13. X	A FIRST preliminary amendment.			
14.	A SECOND or SUBSEQUENT pr	eliminary amendment.		
15.	A substitute specification.			
16.	A change of power of attorney and	l/or address letter.		
17.	A computer-readable form of the se	equence listing in accordance with PCT Rule	13ter.2 and 35 U.S.C. 1.821 - 1.825.	
18.	A second copy of the published int	ternational application under 35 U.S.C. 154(d	1)(4).	
19.	A second copy of the English lange	uage translation of the international application	on under 35 U.S.C. 154(d)(4).	
20. X	Other items or information:			
Abstract			•	
PTO 144	49 with listed references			

JC13 Rec'd PCT/PTO 0 8 MAR 2002

U.S. APPLICATION 10 (19know) To be Assigned	988296 PCT/	TERNATIONAL APPLICATION NO EPOO/08605		ATTORNEY'S DOCK M06994/LeA	
	ing fees are submitted:			CALCULATIONS F	
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Neither internation	al preliminary examinati	on fee (37 CFR 1.482)			
and International Se	arch fee (37 CFR 1.445() earch Report not prepare	a)(2)) paid to USP10 d by the EPO or JPO	\$1040.00		
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but all claims did no	ot satisfy provisions of Po	37 CFR 1.482) paid to US CT Article 33(1)-(4)	\$710.00		
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	ied provisions of PCT A	rticle 33(1)-(4) BASIC FEE AMOU	\$100.00 INT =	\$ 890.00	
				\$ 890.00	
months from the earl	liest claimed priority date	or declaration later than e (37 CFR 1.492(e)).	20 30	\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	14 - 20 =	0	x \$18.00	\$ 84.00	
Independent claims	$\frac{4 - 3}{\text{DENT CLAIM(S) (if approximately fine properties)}}$		x \$84.00 + \$280.00	\$ 84.00 \$	
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are reduced by			+	\$	
		SU	JBTOTAL =	\$	
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		TOTAL NATIO	NAL FEE =	\$ 974.00	
Fee for recording the accompanied by an a	e enclosed assignment (3 appropriate cover sheet (7 CFR 1.21(h)). The assi 37 CFR 3.28, 3.31). \$40.	gnment must be 00 per property +	\$	
		TOTAL FEES E	NCLOSED =	\$ 974.00	
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				charged:	\$
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1.137 (a) or (b)) must be filed and granted to restore the application to pending status				(1)	
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	FAIGHT TRADEMAR		REGISTR	ATION NUMBER	

PATENT APPLICATION Mo-6994 LeA 33,570

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION	ON OF))) DCT/ED00/09605
RÜDIGER G	ORNY ET AL) PCT/EP00/08605)
SERIAL NUMBER: TO BE ASSIGNED)
FILED:	HEREWITH)
TITLE:	POLYCARBONATE SLABS)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

Prior to the examination of the present patent application kindly amend the enclosed translation thereof as follows:

"Express Mail" mailing label numberET671453561US
Date of DepositMarch_8, 2002
I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231 Donna J. Veatch
(Name of person mailing paper or fee) Signature of person mailing paper or fee)
Olganica Sapara

IN THE SPECIFICATION:

On page 1 of the specification, please delete the title and insert the following in its stead:

--POLYCARBONATE SLABS--.

IN THE ABSTRACT:

Please delete the abstract on page 18 and insert the following in its stead:

--POLYCARBONATE SLABS

ABSTRACT OF THE DISCLOSURE

The present invention relates to polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therefore has a dissolved oxygen content of less than 150 ppb.--

A new Abstract page is included herewith.

IN THE CLAIMS:

Cancel Claim 2.

Add the following:

- --9. A sheet comprising polycarbonate, said polycarbonate prepared by the phase interface process that includes an aqueous solution of an alkali salt of a bisphenol that contains dissolved oxygen in an amount of less than 150 ppb.
- 10. A process for preparing a sheet comprising (i) preparing a polycarbonate resin by the phase interface process wherein aqueous solution of an alkali salt of a bisphenol contains dissolved oxygen in an amount of less than 150 ppb (ii) extruding the polycarbonate to form a sheet.
- 11. In the process of making a sheet of polycarbonate resin that is the product of the interface process, the improvement comprising using in said process an aqueous solution of an alkali salt of a bisphenol that contains dissolved oxygen in an amount of less than 150 ppb.

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- 12. The sheet prepared by the process of Claim 10.
- 13. The sheet prepared by the process of Claim 11.
- 14. A method of using the sheet of Claim 12 comprising producing glazing.
- 15. A method of using the sheet of Claim 13 comprising producing glazing.--

REMARKS

The present amendment seeks to place the application in better conformance with U.S. practice.

Entry of the amendment is requested.

Respectfully submitted,

Ву_

Aron Preis Attorney for Applicants Reg. No. 29,426

Bayer Corporation 100 Bayer Road Pittsburgh, Pennsylvania 15205-9741 (412) 777-8343 FACSIMILE PHONE NUMBER: (412) 777-8363

s:/sr/ap0276

VERSION WITH MARKINGS TO SHOW CHANGES MADE:

IN THE SPECIFICATION:

On page 1 of the specification, the title has been deleted and the following inserted in its stead:

--POLYCARBONATE SLABS--.

IN THE ABSTRACT:

The abstract on page 18 has been deleted and the following inserted in its stead:

--POLYCARBONATE SLABS ABSTRACT OF THE DISCLOSURE

The present invention relates to polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therefore has a dissolved oxygen content of less than 150 ppb.--

A new Abstract page is included herewith.

IN THE CLAIMS:

Claim 2 has been cancelled.

The following claims have been added:

- --9. A sheet comprising polycarbonate, said polycarbonate prepared by the phase interface process that includes an aqueous solution of an alkali salt of a bisphenol that contains dissolved oxygen in an amount of less than 150 ppb.
- 10. A process for preparing a sheet comprising (i) preparing a polycarbonate resin by the phase interface process wherein aqueous solution of an alkali salt of a bisphenol contains dissolved oxygen in an amount of less than 150 ppb (ii) extruding the polycarbonate to form a sheet.

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- 11. In the process of making a sheet of polycarbonate resin that is the product of the interface process, the improvement comprising using in said process an aqueous solution of an alkali salt of a bisphenol that contains dissolved oxygen in an amount of less than 150 ppb.
- 12. The sheet prepared by the process of Claim 10.
- 13. The sheet prepared by the process of Claim 11.
- 14. A method of using the sheet of Claim 12 comprising producing glazing.
- 15. A method of using the sheet of Claim 13 comprising producing glazing.--

LeA 33 570-Foreign Countries

-18-

POLYCARBONATE SLABS

ABSTRACT OF THE DISCLOSURE

The present invention relates to polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therefore has a dissolved oxygen content of less than 150 ppb.

- 1 -

Polycarbonate sheets

The present invention relates to polycarbonate sheets, the use thereof to produce glazing and said glazing.

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Polycarbonate sheets are used to produce glazing, for example. In this case, it is desirable to use sheets with as high as possible light transmissions and as low as possible yellowness indexes. Although it is possible to reduce the yellowness index by adding of blue pigments, this is achieved at the expense of light transmission and greying of the sheets. During weathering of the sheets, it is important that the mechanical properties of the sheets are kept at the highest possible level. Even after weathering, the sheets must retain a high resistance to mechanical influences e.g. hail.

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Polycarbonates may be produced by the so-called interfacial polycondensation process. In said process, dihydroxydiarylalkanes (also known as bisphenols) are reacted in the form of their alkali salts in aqueous solution with phosgene in the heterogeneous phase in the presence of inorganic bases such as sodium hydroxide solution and an organic solvent, in which the product polycarbonate is readily soluble.

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More details relating to the interfacial polycondensation process for producing polycarbonates are disclosed in Schnell, "Chemistry and Physics of Polycarbonates", Polymer Reviews, Volume 9, Interscience Publishers, New York, London, Sydney 1964.

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The polycarbonate sheets known from the prior art exhibit the disadvantage that they do not adequately meet the above-mentioned requirements of high light transmission, low yellowness index and good retention of mechanical properties on weathering.

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Donna J. Veatch

(Name of person mailing paper or fee)

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The object of the present invention is therefore to produce polycarbonate sheets which do not exhibit the above-mentioned disadvantages of the prior art.

This object is achieved according to the invention by polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therein has a content of dissolved oxygen of less than 150 ppb.

The content of dissolved oxygen in the aqueous solution of the alkali salt of bisphenol is <150 ppb, preferably <100 ppb, particularly preferably <50 ppb.

The invention thus provides polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therein has a content of dissolved oxygen of less than 150 ppb.

In addition, the present invention also provides glazing, for example for automobiles or buildings, in particular for greenhouses, winter gardens, bus shelters, advertising panels, signboards, protective screens, automotive glazing, windows or roofing.

The polycarbonates are produced according to the invention in that the other raw materials apart from the aqueous solution of the alkali salt of bisphenol do not contain a noteworthy amount of oxygen. In addition, production is performed with the exclusion of oxygen, as described for example in DE-A 4 227 272.

For example, in a reaction vessel and tubular reactor configuration, the forced circulation loop and the tubular reactors are flooded and the reaction vessel is blanketed with nitrogen, to ensure the exclusion of oxygen.

The polycarbonate sheets according to the invention are, for example, solid sheets, hollow sheets (also known as multi wall sheets e.g. twin wall sheets, triple wall

sheets...) or corrugated sheets, which are e.g. used as glazing materials. The sheets may be produced in a manner known in principle to the person skilled in the art, for example by extrusion or by injection moulding.

Subsequent processing of the sheets, such as thermoforming or surface modification, such as application of scratch-proof lacquers, water-dispersing layers and the like, is also possible. The patent also relates to the moulded articles produced by these processes.

To produce the polycarbonate for the sheets according to the invention, aqueous solutions of an alkali salt of a bisphenol with a content of dissolved oxygen <150 ppb, preferably <100 ppb, particularly preferably <50 ppb are used, which may preferably be obtained by reacting bisphenols with a dissolved oxygen content <10 ppb with an aqueous alkali hydroxide solution with a dissolved oxygen content <100 ppb under oxygen exclusion.

Preferred alkali salts are the sodium salts of bisphenols.

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Bisphenols which may be used according to the invention are those which may be obtained by reacting ketones with aromatic hydroxy compounds which are not substituted in the para position and do not contain any second order substituents, such as cyano, carboxy or nitro groups, for example phenol, o- and m-cresol, 2,6-dimethylphenol, o-tert.-butylphenol, 2-methyl-6-tert.-butylphenol, o-cyclohexylphenol, o-phenylphenol, o-isopropylphenol, 2-methyl-6-cyclopentylphenol, o- and m-chlorophenol, 2,3,6-trimethylphenol, preferably phenol, o- and m-cresol, 2,6-dimethylphenol, o-tert.-butylphenol and o-phenylphenol. Phenol is particularly preferred. Ketones with at least one aliphatic group on the carbonyl function are preferred, for example acetone, methyl ethyl ketone, methyl propyl ketone, methyl isopropyl ketone, diethyl ketone, acetophenone, cyclohexanone, cyclopentanone, methyl-, dimethyl- and trimethylcyclohexanones, which may also comprise geminal methyl groups, e.g. 3,3-dimethyl-5-methylcyclohexanone (hydroisophorone).

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Acetone, acetophenone, cyclohexanone and the homologues thereof bearing methyl groups are very particularly preferred. Acetone is the most preferable. By providing an intensive inert nitrogen atmosphere throughout the production process, it is ensured that the residual content of dissolved oxygen in the bisphenols is less than 10 ppb.

Bisphenols which may be used according to the invention are, additionally: 3-(4-hydroxyphenyl)-1,1,3-trimethylindan-5-ol; 1,3-di-(2-(4-hydroxyphenyl)-2-propyl)-benzene and 1,4-di-(2-(4-hydroxyphenyl)-2-propyl)benzene.

Bisphenols which are particularly preferred are 2,2-bis(4-hydroxyphenyl)propane (i.e. bisphenol A) and 1,1-bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane.

The bisphenols or the mixtures thereof are preferably reacted, with the exclusion of oxygen (e.g. by providing an inert nitrogen atmosphere), with aqueous alkali hydroxide solution having a dissolved oxygen content <100 ppb, preferably <20 ppb. The concentration of aqueous alkali hydroxide solution is preferably such that the concentration of the resultant aqueous solutions of an alkali salt of a bisphenol is as close as possible to the solubility limit, i.e. in the range of from 15 to 20 wt.%, preferably 16.5 to 18.5 wt.%. The molar ratio of alkali hydroxide to bisphenol is in particular from 1.8:1 to 2.5:1, preferably 1.9:1 to 2.4:1, particularly preferably 2.0:1 to 2.3:1. The bisphenol may be dissolved as a solid in aqueous alkali hydroxide solution. However, it is preferable for it to be added to the aqueous alkali hydroxide solution directly as a melt at temperatures of from 20°C to 90°C, preferably 30°C to 70°C, without its having to pass through the solid state.

The virtually oxygen-free aqueous alkali hydroxide solution used to produce the aqueous solutions of an alkali salt of a bisphenol may be produced by electrolysis. After production, the aqueous alkali hydroxide solution should be stored and transported under inert gas. For use in the process according to the invention, the concentration of aqueous alkali hydroxide solution obtained during electrolysis is

generally reduced by dilution with virtually oxygen-free, fully deionised water. Oxygen is removed from the fully deionised water in a manner known in principle, e.g. catalytically, by degassing or by inert gas stripping.

The aqueous solutions thus obtained of an alkali salt of a bisphenol exhibit particularly low colour indexes, which are naturally also dependent on the colour index of the bisphenol used. If a bisphenol is used which has a colour index <10 Hazen (measured according to ASTM D 1686), colour indexes of <1.5 Hazen, preferably <1.0 Hazen may be achieved.

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The polycarbonates may be branched deliberately and in a controlled manner by the use of small amounts of branching agent. Examples of suitable branching agents are: phloroglucinol, 4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)-2-heptene; 4,6-dimethyl-2,4,6-tri-(4-hydroxyphenyl)heptane; 1,3,5-tri-(4-hydroxyphenyl)benzene; 1,1,1-tri-(4-hydroxyphenyl)ethane; tri-(4-hydroxyphenyl)phenylmethane; 2,2-bis-[4,4-bis(4-hydroxyphenyl)cyclohexyl]propane; 2,4-bis(4-hydroxyphenylisopropyl)phenol; 2,6-bis(2-hydroxy-5'-methylbenzyl)-4-methylphenol; 2-(4-hydroxyphenyl)-2-(2,4-dihydroxyphenyl)propane; hexa-(4-(4-hydroxyphenylisopropyl)phenyl)orthoterephthalic acid ester; tetra-(4-hydroxyphenyl)methane; tetra-(4-(4-hydroxyphenylisopropyl)phenoxy)methane); α,α',α'' -tris-(4-hydroxyphenyl)-1,3,5-triisopropylbenzene; 2,4-dihydroxyphenyl)-2-oxo-2,3-dihydroindole; 1,4-bis(4',4"-dihydroxytriphenyl)methyl)-benzene. 1,1,1-tri-(4-hydroxyphenyl)ethane and bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2,3-dihydroindole (= isatinbiscresol).

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The branching agents or mixtures of branching agents which may optionally also be used, preferably in an amount of 0.05 to 2 mol-% relative to the bisphenols used, may be introduced together with the bisphenols or added at a later stage of the synthesis.

Chain terminators may be used according to the invention. The chain terminators used according to the invention are preferably phenols such as phenol, alkylphenols such as cresol and 4-tert.-butylphenol, chlorophenol, bromophenol, cumylphenol or mixtures thereof. Phenol, 4-tert.-butylphenol or cumylphenol are particularly preferred.

Chain terminators and branching agents may be added to the reaction mixture as a separate solution or together with the bisphenolate, wherein care must be taken to ensure that the solutions have the same low oxygen content according to the invention as the aqueous solutions of an alkali salt of a bisphenol.

Other additives which may be used according to the invention and which may be added to the polycarbonate from which the sheets according to the invention are produced, are mentioned in EP-A 0 500 496 and WO 96/15102. According to the invention, mixtures of these additives may also be introduced into the polycarbonate.

Additives which are particularly preferred are UV absorbers such as the compounds described in WO 99/05205 of formula (I)

$$(R_1)_n$$
 $(R_1)_n$
 $(R_1)_n$
 $(R_2)_m$
 $(R_2)_m$
 $(R_2)_m$
 $(R_2)_m$
 $(R_3)_m$
 $(R_4)_m$
 $(R_2)_m$

in which

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 R^1 and R^2 are the same or different and mean H, halogen, C_1 - C_{10} alkyl, C_5 - C_{10} cycloalkyl, C_7 - C_{13} aralkyl, C_6 - C_{14} aryl, $-OR^5$ or -(CO)- $O-R^5$, with R^5 being H or C_1 - C_4 alkyl,

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 R^3 and R^4 are also the same or different and mean H, $C_1\text{-}C_4$ alkyl, C_6 cycloalkyl, benzyl or $C_6\text{-}C_{14}$ aryl,

m is 1, 2 or 3 and

n is 1, 2, 3 or 4,

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and also those of formula (II)

$$(R_1)_n$$
 $(R_1)_n$
 $(R_1)_n$
 $(R_2)_n$
 $(R_2)_n$
 $(R_2)_n$
 $(R_3)_n$
 $(R_4)_n$
 $(R_2)_n$
 $(R_3)_n$

in which the bridge means

$$---(CHR^3)_{p}$$
 $---(CHR^4)_{q}$ $---(CHR^4)_{p}$ $----(CHR^4)_{p}$

R¹, R², m and n have the meaning stated in relation to formula (I),

in which, moreover, p is a whole number from 0 to 3,

q is a whole number from 1 to 10,

Y is -CH₂-CH₂-, -(CH₂)₃-, -(CH₂)₄-, -(CH₂)₅-, -(CH₂)₆-, or CH(CH₃)-CH₂

and

R³ and R⁴ have the meaning stated in relation to formula (I).

Other suitable UV absorbers are those comprising substituted triazines, such as 2,4-bis(2,4-dimethylphenyl)-6-(2-hydroxy-4-n-octyloxyphenyl)-1,3,5-triazine (CYASORB® UV-1164) or 2-(4,6-diphenyl-1,3,5-triazin-2-yl)-5-(hexyl)oxyphenol (Tinuvin® 1577). The UV absorber which is particularly preferred is 2,2-methylene bis(4-(1,1,3,3-tetramethylbutyl)-6-(2H-benzotriazol-2-yl)phenol), which is sold commercially under the names Tinuvin® 360 or Adeka Stab® LA 31. The UV absorber Uvinul 3030, made by BASF AG and obtained in accordance with Example 1 of WO 96/15102, is also suitable, as are the UV absorbers given in EP 0 500 496 A1.

Other suitable UV absorbers are 5'-methyl-, 3',5'-di-tert.-butyl-, 5'-tert.-butyl-, 5'-(1,1,3,3-tetramethylbutyl)-, (= Tinuvin 329), 5-chloro-3',5'-di-tert.-butyl-, 5-chloro-3'-tert.-butyl-5'-methyl-, 3'-sec.-butyl-5'-tert.-butyl, 4'-octoxy-, 3',5'-di-tert.-amyl-, 3',5'-bis(α , α -dimethylbenzyl)-, mixture of 5-chloro-3'-tert.-butyl-5'-(2-octyloxycarbonylethyl)- and 5-chloro-3'-tert.-butyl-5'-[2-(2-ethylhexyloxy)carbonylethyl]-, 5chloro-3'-tert.-butyl-5'-(2-methoxycarbonylethyl)-, 3'-tert.-butyl-5'-(2-methoxycarbonylethyl)-, 3'-tert.-butyl-5'-(2-octyloxycarbonylethyl)-, 3'-tert.-butyl-5'-[2-(2-ethylhexyloxy)-carbonylethyl]-, 3'-dodecyl-5'-methyl- and 3'-tert.-butyl-5'-(2-isooctyl-2,2'-methylenebis[4oxycarbonylethyl)-2'-hydroxyphenyl-2H-benzotriazole(2), (1,1,3,3-tetramethylbutyl)-6-benzotriazol-2-yl-phenol] together with 3'-sec.-butyl-5'-(1,1,3,3-tetramethylbutyl-2'-hydroxyphenyl-2H-benzotriazole (= Tinuvin 350) and 2-[2'-hydroxy-3',5'-bis(1,1-dimethylbenzyl)phenyl]-2H-benzotriazole (= Tinuvin 234) and 2-hydroxybenzophenones, such as for example 4-hydroxy-, 4-methoxy-, 4octoxy-, 4-decyloxy-, 4-dodecyloxy-, 4-benzyloxy-, 4,2',4-trihydroxy-, 2'-hydroxy-4,4'-dimethoxy derivative, ethyl-2-cyano-3,3-diphenyl acrylate (= Uvinul 3035) made by BASF AG, 2-ethylhexyl-2-cyano-3,3-diphenyl acrylate (= Uvinul 3039) made by BASF AG.

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Examples of suitable stabilisers, which may be used as additives according to the invention, are phosphines, phosphites or Si-containing stabilisers and other compounds described in EP-A 0 500 496. Triphenyl phosphites, diphenylalkyl phosphites, phenyldialkyl phosphites, tris(nonylphenyl) phosphite, triphenyl phosphine, tetrakis(2,4-di-tert.-butylphenyl)-4,4'-biphenylene diphosphonite and triarylphosphite may be cited by way of example. Triphenylphosphine and tris(2,4-di-tert.-butylphenyl)phosphite are particularly preferred.

The polycarbonates, from which the sheets according to the invention are produced, may additionally contain additives to improve the surface slip characteristics, for example esters of polyhydric alcohols with long-chain fatty acids, such as for example pentaerythritol tetrastearate/-palmitate, glycerol monostearate/-palmitate, glycerol tristearate/-palmitate. Furthermore, the addition of flame retardants, pigments, colorants, finely divided minerals, optical brighteners and other additives is also possible.

All the feed materials and solvents used for synthesis may be contaminated with impurities from the production and storage thereof, wherein the aim is to work with starting materials which are as clean as possible.

Glazing according to the invention comprises, in particular, glazing for automobiles or buildings, for example glazing for greenhouses, winter gardens, bus shelters, advertising panels, signboards, protective screens, automotive glazing, windows or roofing.

Subsequent processing of the sheets according to the invention, such as thermoforming or surface modification, such as application of scratch-proof lacquers, water-dispersing layers and the like, is possible.

Examples

In the following Examples, the colour indexes were determined according to ASTM D 1686 by measuring absorption up to 400 nm over an irradiation length of 50 cm.

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Weathering of these sheets (according to ISO 4892-2A) was performed in a Weatherometer made by Atlas, USA, using a 6.5 W xenon lamp and a cycle involving 102 mins of exposure to light and 18 mins of spraying with deionised water together with light exposure. The maximum black body temperature was 60°C (± 5°C). After 1000 hours, samples of the sheets were taken and toughness was tested under practical conditions using a modified falling dart test which was developed for multi-walled sheets on the basis of DIN 53 443. The fracture energy W_s being measured using a 5 mm diameter puncture body intended to cause biaxial stress similar to that caused by a falling hailstone. At room temperature, a falling mass of 36 kg falling from a height of 0.2 m struck the samples, which lay freely on a supporting ring with an internal diameter of 20 mm.

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Testing was performed with the weathered side of the sample in the compression zone. The fracture appearance (BB) was characterised by numbers: 1 = splintered, 2 = smooth crack, 3 = ductile/brittle, 4 = ductile.

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The yellowness index determined according to ASTM E 313 was stated as Yellowness Index YI.

Example 1

To produce 1.022 t/h of a 15% aqueous NaBPA solution, 867.5 kg/h of a 6.5% aqueous sodium hydroxide solution, and 154.5 kg/h of bisphenol A melt were combined continuously. The entire process was performed under an inert atmosphere of nitrogen. The 6.5% aqueous sodium hydroxide solution comprised an oxygen content of 10 ppb. The Hazen colour index of the resultant 15% aqueous sodium bisphenolate solution amounted to 0.5 Hazen. This sodium bisphenolate solution was used to produce polycarbonate by the interfacial polycondensation process.

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Comparative Example 2

To produce 1.022 t/h of a 15 % aqueous sodium bisphenolate solution, 867.5 kg/h of a 6.5% aqueous sodium hydroxide solution and 154.5 kg/h of bisphenol A melt were combined continuously. The entire process was performed under an inert atmosphere of nitrogen. The 6.5% aqueous sodium hydroxide solution comprised an oxygen content of 250 ppb. The Hazen colour index of the resultant 15% aqueous sodium bisphenolate solution amounted to 2 Hazen. This sodium bisphenolate solution was used to produce polycarbonate by the interfacial polycondensation process.

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Example 3

A polycarbonate with phenol end groups, a melt flow index (MFR) of 3 (measured according to ISO 1133), a branching agent content of 0.3 mol% isatinbiscresol and a UV absorber content of 0.25% Tinuvin® 350 (3'-sec.-butyl-5'-(1,1,3,3-tetramethyl-butyl-2'-hydroxyphenyl-2H-benzotriazole) was produced from the sodium bisphenolate solution obtained in Example 1. This polycarbonate was processed in the following plant to produce 10 mm coextruded twin-wall sheets:

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main extruder having a screw of length 33D and a diameter of 70 mm with venting

- coextruder for applying the outer layer having a screw of length 25D and a diameter of 30 mm
- special sheet die with a width of 350 mm
- calibrator
- 5 roller conveyor
 - take-off unit
 - cutting device (saw)
 - stacking table.
- The polycarbonate obtained from Example 1 was fed introduced into the coextruder.

 Coextrusion as such is known in the art (see for example EP-A 110 221 and EP-A 110 238). In the present instance, the process was performed as follows:
- Extruders for producing the core layer and the outer layer(s) were connected to a coextrusion adapter. The adapter was designed that the melt forming the outer layer was applied as a thin layer adhering to the core layer melt.
 - The multi-layer melt strand thereby produced was then given the desired form (twin-walled sheet) in the die connected downstream. The melt was then cooled in a known way under controlled conditions by means of vacuum calibration and subsequently sawn into pieces 1 metre in length.

Comparative Example 4

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A polycarbonate with phenol end groups, a melt flow index (MFR) of 3 (measured according to ISO 1133), a branching agent content of 0.3 mol% isatinbiscresol and a UV absorber content of 0.25% Tinuvin® 350 (3'-sec.-butyl-5'-(1,1,3,3-tetramethyl-butyl-2'-hydroxyphenyl-2H-benzotriazole) was produced from the sodium bisphenolate solution obtained in Example 2. This polycarbonate was processed in the same plant and under the same conditions as in Example 3 to produce 10 mm coextruded twin-wall sheets.

Example 5

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A polycarbonate with phenol end groups and a melt flow index (MFR) of 3 (according to ISO 1133) was produced from the sodium bisphenolate solution obtained in Example 1. This polycarbonate was processed in the same plant and under the same conditions as in Example 3 to produce 10 mm coextruded twin-wall sheets.

A compound of the polycarbonate obtained from Example 1 and 5% of a benzo-triazole UV absorber (Tinuvin® 360 = 2,2-methylenebis-(4-(1,1,3,3-tetramethyl-butyl)-6-(2H-benzotriazol-2-yl)phenol) was introduced into the coextruder. This compound was produced in the following manner: the UV absorber was mixed into the polycarbonate in a twin-screw extruder (ZSK 32, Werner & Pfleiderer) at 280°C and 80 rev/min and the extrudate was then pelletised.

Comparative Example 6

A polycarbonate with phenol end groups and a melt flow index (MFR) of 3 (according to ISO 1133) was produced from the sodium bisphenolate solution obtained in Example 2. This polycarbonate was processed in the same plant and under the same conditions as in Example 3 to produce 10 mm coextruded double-wall sheets.

A compound of the polycarbonate obtained from Example 1 and 5% of a benzotriazole UV absorber (Tinuvin® 360 = 2,2-methylenebis-(4-(1,1,3,3-tetramethylbutyl)-6-(2H-benzotriazol-2-yl)phenol) was introduced into the coextruder. This compound was produced in the following manner: the UV absorber was mixed into the polycarbonate in a double-shaft extruder (ZSK 32, Werner & Pfleiderer) at 280°C and 80 rev/min and the extrudate was then pelletised.

Example 7

A polycarbonate with phenol end groups, a melt flow index (MFR) of 3 (measured according to ISO 1133) and a UV absorber content of 0.3% Tinuvin® 350 (3'-sec.-butyl-5'-(1,1,3,3-tetramethylbutyl-2'-hydroxyphenyl-2H-benzotriazole) was produced from the sodium bisphenolate solution obtained in Example 1. This polycarbonate was processed by injection moulding to produce colour chips 3 mm thick.

Comparative Example 8

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A polycarbonate with phenol end groups, a melt flow index (MFR) of 3 (measured according to ISO 1133) and a UV absorber content of 0.3% Tinuvin® 350 (3'-sec.-butyl-5'-(1,1,3,3-tetramethylbutyl-2'-hydroxyphenyl-2H-benzotriazole) was produced from the sodium bisphenolate solution obtained in Example 2. This polycarbonate was processed by injection moulding to produce colour chips 3 mm thick.

The sheets obtained were then subjected to colorimetric assessment, using the following measuring methods:

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- Transmission (on the basis of standard ASTM D 1003)

 Apparatus: Pye-Unicam (measuring geometry: 0°/diffuse, calculated according to illuminant C)
- 2. Yellowness index YI according to ASTM E 313

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Weathering of these sheets was performed according to ISO 4892-2A in the Weatherometer made by Atlas, USA using a 6.5 W xenon lamp and a cycle involving 102 mins of exposure to light and 18 mins of spraying with deionised water together with light exposure. The maximum black body temperature was 60°C (± 5°C). After 1000 hours, samples of the sheets were taken and toughness was tested under practical conditions using a modified

falling dart test developed for multi wall sheets on the basis of DIN 53 443, the fracture energy W_s being measured using a 5 mm diameter puncture body intended to cause biaxial stress similar to that caused by a falling hailstone. At room temperature, a falling mass of 36 kg falling from a height of 0.2 m struck the samples, which lay freely on a supporting ring with an internal diameter of 20 mm.

Testing was performed with the weathered side of the sample in the compression zone. The fracture appearance (BB) was characterised by numbers: 1 = splintered, 2 = smooth crack, 3 = ductile/brittle, 4 = ductile.

The yellowness index determined according to ASTM E 313 was stated as Yellowness Index YI.

15 Results:

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	Transmission (%)	Yellowness Index
Example 3	79.5	1.2
Comparative Example 4	77.4	2.7
Example 7	87.3	3.0
Comparative Example 8	84.0	5.5

	Appearance of fracture (0 hours weathering as stated)	Appearance of fracture (4000 hours weathering as stated)	Appearance of fracture (5000 hours weathering as stated)
Example 3	4x4	4x4	4x4
Comparative example 4	4x4	4x3	4x3
Example 5	4x4	4x4	4x4
Comparative example 6	4x4	3x4, 1x3	3x4, 1x3

The results show that sheets produced according to the invention have higher transmission values together with lower Yellowness Index values and better mechanical stability after weathering than the sheets according to comparative tests.

Claims

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- 1. Polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therefore has a dissolved oxygen content of less than 150 ppb.
- 2. Use of the sheets according to claim 1 to produce glazing.
- 3. Glazing comprising sheets according to claim 1.
 - 4. Glazing for automobiles or for buildings or for greenhouses or for wintergardens or for bus shelters or for advertising panels or signboards or protective screens or for windows or roofing comprising sheets according to claim 1.
 - 5. Multi-layered sheet, wherein at least one layer contains the polycarbonate according to claim 1.
- 20 6. Proces for the production of the sheets according to claim 1 by extrusion.
 - 7. Process for the production of the multi-layered sheets according to claim 5 by co-extrusion.
- Multi-layered sheets according to claim 5, wherein at least one layer contains UV-Absorber, the concentration of the UV-Absorber in the layer being preferably 0.01 to 20 % by weight.

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Polycarbonate sheets

Abstract

The present invention relates to polycarbonate sheets, wherein the polycarbonate is produced by the interfacial polycondensation process and the aqueous solution of an alkali salt of a bisphenol used therefore has a dissolved oxygen content of less than 150 ppb.

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COMBINED DECLARATION AND POWER OF ATTORNEY

APR 1 5 2002

that: As a below named inventor, I hereby declare

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is

on the invention entitled

POLYCARBONATE SLABS

the specification of which is attached hereto,

or was filed on September 4, 2000

as a PCT Application Serial No. PCT/EP00/08605

I hereby state that I have reviewed and understand the contents of the aboveidentified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, \$119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

199 43 640.1 (Number)

Germany (Country) September 13, 1999 (Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, \$120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, \$112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, \$1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Le A 33 570-US

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